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THE ROLE OF THE INTERFACE ON THE MECHANICAL BEHAVIOR OF METAL M--ETC(U)  
JAN 79 A LAWLEY

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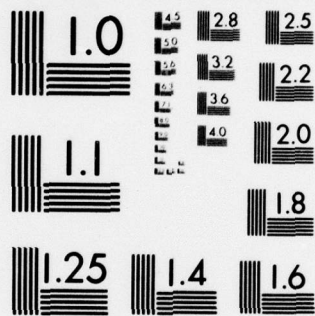
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6 THE ROLE OF THE INTERFACE ON THE MECHANICAL  
BEHAVIOR OF METAL MATRIX COMPOSITES.

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Principal Investigator: A. Lawley

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ABSTRACT

The microstructural stability and mechanical behavior of the in-situ Co,Cr-(Cr,Co) $\frac{1}{3}$ C $\frac{1}{3}$  composite and filamentary Ni-Mo and nichrome-tungsten composites have been characterized as a function of isothermal exposure and various regimes of thermal cycling. Properties examined were strength, toughness and resistance to fatigue crack propagation. Correlations were developed between microstructural change accompanying each mode of heat-treatment and the above properties. This provided the basis for the development of models for plastic flow and crack initiation/propagation in the two types of composite material which can account for the observed effect(s) of the heat-treatment imposed.

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## 1. INTRODUCTION

Metal matrix composites possess attractive property combinations for current and projected Navy material requirements and areas of application. High strength-to-ductility, stiffness-to-density, and reasonable ductility/toughness, are compatible with marine and aircraft structural elements. With a superalloy matrix, creep resistance and stress rupture, coupled with oxidation and corrosion resistance, enhance viability as a material for blades/vanes in advanced aircraft engines and land-based turbines.

Integrity of performance under load is vested in the elastic/plastic response of the composite, this being controlled directly by the interface between matrix and reinforcement. The interface or interfacial region between constituent phases of the composite is the medium across which stress is transferred from matrix to reinforcement. Thus, the nature, structure, and stability of the interface are key factors in attempting to: a) predict the response under load, and b) optimize the composite microstructure, in a given application, knowing the attendant service conditions.

Two distinct forms of interface may be identified in metal-matrix composites namely: (i) the interface characteristic of directionally solidified (in-situ) composites, and (ii) the interface in a filamentary wire or fiber-reinforced matrix fabricated by techniques such as diffusion bonding or liquid metal infiltration.

In directionally solidified composites, constituent phases are in chemical equilibrium so that the microstructure is expected to be stable up to high homologous temperatures. This has been confirmed in several eutectic systems having a lamellar or rod-like morphology of the reinforcing phase. While the level of microstructural stability is high, 'physicochemical instability' does promote coarsening/spheroidization during thermal cycling or prolonged isothermal exposure at homologous temperatures  $\geq 0.9 T_{\text{eutectic}}$ . More recently, it has also

been demonstrated that microstructural instability and other forms of damage are associated with thermal cycling of aligned eutectics.

Differences in chemical potential at matrix-reinforcement interfaces in filamentary composites are the basis of 'chemical' instability. Thus, this form of composite is prone to chemical reaction and/or diffusion at the interface with accompanying solid solution alloying and/or compound formation. The effect has been characterized in a spectrum of composites of this type for thermal cycling or isothermal exposure. Unlike coarsening of in-situ composites, chemical instability is accompanied by changes in composition and volume fraction of reinforcement.

## 2. PROGRAM OBJECTIVES

Microstructural instability is accompanied by changes in mechanical properties and in order to realize the full potential of either form of composite, a detailed and quantitative structure property correlation is essential - with particular reference to the role of the interface. This has been the primary goal of this program. Properties examined included strength, toughness, and fatigue resistance; these were determined in the as-grown or as-fabricated condition of the composite and following various regimes of high temperature isothermal exposure or thermal cycling.

## 3. COMPOSITE SYSTEMS

The in-situ composite selected was  $\text{Co,Cr-(Cr,Co)}_7\text{C}_3$ . A eutectic is formed by a monovariant ternary reaction and contains 30% by volume of the aligned fibrous carbide in a cobalt-rich matrix. The nature of the constituents suggest that the alloy might be useful as an elevated temperature structural

material; the composite exhibits high strength and creep resistance and good corrosion/oxidation resistance but limited room temperature ductility in the as-grown condition.

Most of the work on the filamentary type of composite was on Ni-Mo. While this is a model high-temperature system, alloys of the constituents are candidate materials for gas turbine applications. By testing above and below the DBTT of the reinforcement, a direct comparison of the tensile response of the ductile matrix-ductile fiber and ductile matrix-brittle fiber forms was possible for a single fiber material. Some comparative studies were also carried out on the nichrome-tungsten system with and without yttrium oxide as a diffusion barrier coating at the interface.

#### 4. EXPERIMENTAL PROCEDURES

Details of the various procedures developed in this program are given in the pertinent ONR Technical Reports listed at the end of this report. A brief summary is given here.

In-situ composites of  $\text{Co,Cr-(Cr,Co)}_7\text{C}_3$  were directionally solidified at rates up to  $47.6 \times 10^{-6}$  m/s to give an aligned reinforcement of  $(\text{Co,Cr})_7\text{C}_3$  in the cobalt-rich matrix. To determine toughness, the work-of-fracture test-piece configuration was used. Fatigue crack propagation (FCP) response was characterized using a four-point bend configuration specimen. Isothermal exposures were at temperatures up to  $T/T_m = 0.92$ . A spectrum of thermal cycling regimes was examined to include hold times at  $T_{\text{max}}$ .

Filamentary composites of Ni-Mo and nichrome-tungsten were fabricated by diffusion bonding. Subsize Charpy impact specimens were used to evaluate toughness. The Charpy machine was instrumented to display striker velocity, load and energy absorbed. Isothermal exposure was carried out at 1100°C.

## 5. SUMMARY OF RESULTS

### Co,Cr-(Cr,Co)<sub>7</sub>C<sub>3</sub> In-Situ Composite

#### a) Elevated Temperature Stability

To assess microstructural stability, composites were subjected to isothermal exposures at homologous temperatures of 0.85, 0.89 and 0.92 respectively, for times up to  $2.5 \times 10^6$  s. Transverse sections were examined as a function of (T,t) by a combination of optical and scanning electron microscopy and provided the basis for a detailed and quantitative evaluation of changes in rod density (number per unit area), rod spacing, spread of spacings, rod shape and shape spread. While the observations confirm good high-temperature stability, changes in microstructure occurred with an associated decrease in rod density. The faceted appearance of the carbide rods, characteristic of the as-grown condition, gave way to a more rounded appearance. Subtle manifestations of structural instability exist which are related to the irregular cross-sectional form of the rod carbides in the as-grown composite. Data have been analyzed in terms of the competing coarsening mechanisms of two-dimensional ripening, fault migration and fault migration plus annihilation.

#### b) Fatigue Crack Propagation

Fatigue crack propagation has been characterized at room temperature in the as-grown condition and following post solidification isothermal exposure or thermal cycling. The dependence of  $da/dN$  on  $\Delta K$  follows the Paris-Erdogan relation over the range  $\Delta K \sim 10 \text{ MN.m}^{-3/2}$  to  $\sim 50 \text{ MN.m}^{-3/2}$ . Fracture toughness is low in the as-grown condition and is attributed to restricted matrix slip and a low stacking fault energy in the cobalt-rich matrix coupled with the absence of delamination. The heat treatments enhance fracture toughness and this is shown to be the result of precipitation of  $(\text{Co,Cr})_{23}\text{C}_6$  in the matrix and/or fiber coarsening with an attendant increase in the interfiber

spacing. At the lower end of the  $\Delta K$  range fatigue cracking is primarily crystallographic (stage I) in nature. Stage II cracking, with the fracture surface normal to the stress axis, is operative at the upper end of the  $\Delta K$  range; the transition occurs between  $17 \text{ MN.m}^{-3/2}$  and  $28 \text{ MN.m}^{-3/2}$ . The fracture toughness of  $\text{Co,Cr-(Cr,Co)}_7\text{C}_3$  is inferior to  $\text{CoTaC}$  or the lamellar  $\gamma/\gamma'-\delta$  and  $\gamma-\delta$  in-situ composites. These differences are rationalized in terms of constituent matrix and fiber properties.

c) Toughness

Work of fracture has been determined at room temperature in the as-grown condition and following post-solidification isothermal exposure or thermal cycling. Toughness is low in the as-grown condition and is attributed to restricted matrix slip and a low stacking fault energy in the cobalt-rich matrix coupled with the absence of crack deflection and matrix-interface delamination. In general, the heat-treatments did not lead to any major deterioration in toughness, rather in some cases toughness was enhanced by a factor of about two over that in the as-grown composite. Changes in toughness after isothermal exposure are attributed to microstructural changes involving degeneration of the  $(\text{Cr,Co})_7\text{C}_3$  into a precipitate of  $(\text{Cr,Co})_{23}\text{C}_6$  and to fiber coarsening with an attendant increase in interfiber spacing and fiber diameter. In thermal cycling, thermal fatigue and fiber degradation are superimposed on degeneration and coarsening. A model based on thermal residual stresses and strains resulting from thermal expansion mismatch of matrix and fiber has been developed; experimental results for the two cycling regimes examined are in good agreement with the model. The increase in toughness is proportional to the temperature range of the cycle; cycles involving long times above the matrix relaxation temperature lead to creep/recovery of the matrix accompanied by a decrease in toughness.

d) Oxidation

Some preliminary observations have been made on the oxidation response of  $\text{Co,Cr-(Cr,Co)}_7\text{C}_3$  in air at  $1121^\circ\text{C}$ . These include weight gain and subsequent room temperature strength, hardness and toughness. The composite shows superior oxidation resistance to several other in-situ composites by virtue of its high chromium content. Oxidation enhances toughness but leads to a decrease in hardness and strength. The toughness increase is associated with fiber coarsening.

e) Strength

The effect of elevated temperature isothermal exposure and thermal cycling on the microstructural stability and compressive strength of the in-situ rod-like composite  $\text{Co,Cr-(Cr,Co)}_7\text{C}_3$  ( $V_f \approx 0.3$ ) has been examined. Cycling regimes studied were  $357^\circ\text{C}$  to  $913^\circ\text{C}$ ,  $534^\circ\text{C}$  to  $1121^\circ\text{C}$  and  $736^\circ\text{C}$  to  $913^\circ\text{C}$  for up to  $10^4$  cycles with and without hold time at  $T_{\max}$  and  $T_{\min}$ . Isothermal exposure temperatures were  $913^\circ\text{C}$ ,  $1121^\circ\text{C}$  and  $1242^\circ\text{C}$  ( $T_m = 1303^\circ\text{C}$ ) for periods up to  $3.7 \times 10^6$  s. Microstructures were characterized by optical and electron metallography, x-ray point analysis and electron diffraction. Strength was evaluated in compression at ambient and elevated temperatures.

Significant changes in microstructure accompany each of the above heat-treatments and in general strength increases. This is caused by a combination of precipitation and dissolution of  $(\text{Co,Cr})_{23}\text{C}_6(\text{K}'_3)$ , rounding and splitting of the primary  $(\text{Co,Cr})_7\text{C}_3(\text{K}'_2)$  fibers, spheroidization of  $\text{K}'_2$  and/or  $\text{K}'_3$  and void formation at fiber-matrix interfaces. The observed strength changes are in good agreement with those calculated from the Orowan-Ashby model of dispersion

hardening and a modified Hall-Petch relationship in which the inter-fiber spacing is analogous to the grain diameter.

Failure in compression below the ductile to brittle transition temperature of the reinforcement ( $\approx 593^{\circ}\text{C}$ ) occurs by shear in the matrix accompanied by transverse cleavage of the fibers in the "in phase" buckling mode. The overall plane of fracture is oriented at  $45^{\circ}$  to the axis of compression. Secondary cracks, voids and elongated dimples reflect ductile matrix shear. Above the ductile-to-brittle transition temperature of the reinforcing phase ( $K_2'$ ) both matrix and carbide exhibit plastic flow. Intensive shear bands give rise to fiber break-up and fragmentation. Kink bands are the result of elastic buckling of the fibers. Voids and debonding do not occur at the fiber-matrix interface. However, above  $1121^{\circ}\text{C}$  the Co-rich matrix softens and dynamic recovery occurs accompanied by interfacial delamination parallel to the axis of compression.

## Ni-Mo and Nichrome-Tungsten Filamentary Composites

### a) Strength

The tensile behavior of Ni-Mo and Nichrome-W filamentary composites ( $V_f \leq 0.2$ ) was examined with particular reference to the effect of prolonged exposure at temperatures up to 1473°K. Composites were tested above and below the DBTT of the wire reinforcement to give a direct comparison of ductile matrix-ductile fiber and ductile matrix-brittle fiber behavior. Data were analyzed in terms of micromechanical models of composite deformation. For ductile fiber reinforcement, the rule of mixtures analysis of composite tensile strength must allow for a reduction in  $V_f$  due to interface reaction, and for a decrease in the fracture strain of the composite compared to the as-fabricated condition. With brittle reinforcement, following elevated temperature exposure, composite tensile strength was lower than that of the matrix material; fiber-matrix reaction induces notch sensitivity in the fiber and alters constituent properties such that the fiber content required for reinforcement is raised to a level above that existing in the nickel or nichrome matrix. The nature and extent of the interface-reaction products were characterized and the effectiveness of diffusion barrier coatings of  $Y_2O_3$  appraised.

### b) Toughness

The correlation between toughness, microstructure, and interface form was examined for nickel-molybdenum composites ( $V_f$  0.08 and 0.20) in the diffusion-bonded condition and following elevated-temperature exposure. Instrumented Charpy tests were conducted on subsize specimens notched perpendicular to the direction of reinforcement. By testing above (ambient) and below the ductile to brittle transition temperature (DBTT) of the wire reinforcement, a direct comparison of impact response of ductile matrix-ductile

fiber and ductile matrix-brittle fiber systems was possible. Composites exhibited a high level of toughness for all combinations of  $V_f$ , exposure history, and impact temperature; Charpy values were in the range 28-36 ft. lbs. (39-48.7J) and 10-19 ft.lbs. (13.5-25.7J) for  $V_f$  levels of 0.08 and 0.20, respectively. For each combination of test temperature and composite structure, increasing the level of reinforcement lowered toughness. Wire necking occurred at both  $V_f$  levels in the as-fabricated condition at room temperature. Pullout was restricted to the lower volume fraction composites. Multiple fiber fracturing was a characteristic feature of low-temperature impact testing. Impact energy for the ductile matrix-brittle fiber condition can be predicted from the Cooper-Kelly model if an effective flow-stress is used for the matrix to accommodate work-hardening. A micromechanical model was developed for the ductile fiber condition which allowed for prediction of impact energy from tensile behavior of the constituents, and which accounted for the change in toughness associated with interface intermetallic compounds.

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"Effect of Thermal Treatment on the Structure and Toughness of the Co,Cr-(Cr,Co)<sub>7</sub>C<sub>3</sub> In-Situ Composite" (December 1978; M. H. Abdellatif and A. Lawley).

"Fatigue Crack Propagation in a Co,Cr-(Cr,Co)<sub>7</sub>C<sub>3</sub> Composite" (December 1978; M. H. Abdellatif and A. Lawley).

"Effect of Oxidation on the Toughness and Strength of the Co,Cr-(Cr,Co)<sub>7</sub>C<sub>3</sub> In-Situ Composite" (January 1979; M. H. Abdellatif and A. Lawley).

"Effect of Elevated Temperature Exposure and Thermal Cycling on the Strength of Co,Cr-(Cr,Co)<sub>7</sub>C<sub>3</sub> In-Situ Composites" (January 1979; H. Saatchi and A. Lawley).

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